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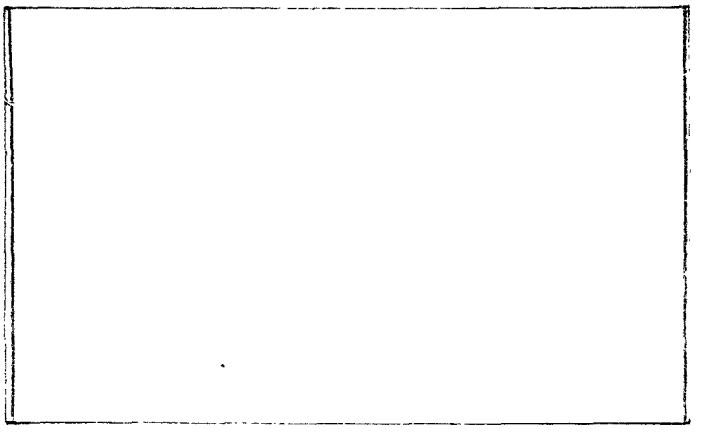



*LEAR SIEGLER, INC.*

*RESEARCH LABORATORIES*

*SANTA MONICA, CALIFORNIA*

# SEMICONDUCTOR THIN FILMS

CONTRACT NO. AF33(657)-7623

Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio

QUARTERLY REPORT NO. 5
November 1, 1962 to March 31, 1963

LIST OF ILLUSTRATIONS

## 1.0 PREVIOUS PLANS FOR FIFTH PERIOD

1.1 To construct a small glass system in which ultra high vacuum would be obtained by gettering or cryosorption.

1.2 To investigate the effect of annealing on GaAs films.

1.3 To make Hall mobility measurements at room temperature and to set up for low temperature measurement.

1.4 To deposit GaAs on BeO and to continue the search for $CeO_2$ and other substrates.

1.5 To continue improvement of electron microscopy techniques.

1.6 To continue improvement of the GaAs evaporation source.

1.7 To investigate deposition at microns per second.

1.8 To continue device exploration using films of available quality.



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2.0 ACCOMPLISHMENTS

2.1 The tube source shown in Figure 1 of Quarterly Report No. 4 (QR4) gave thinner films for a given amount of feed than did an open boat. A new source was built (Figure 1) which gave deposits about as thick per gram evaporated as did the boat but with much less powder sticking to the substrate.

2.2 Hall measurements were made using the circuit shown on page 5 of QR4.

| Sample | Type | r ohm cm | R $cm^3$/coul. | $\mu$ $cm^2$/vs | T °C |
|---|---|---|---|---|---|
| J546-2P | p | 4.5 | 21 | 4.6 | room |
| J556B | p | 2.5 | 63 | 25 | room |
| J549A | p+ | 0.6 | 34 | 61 | room |
| J552B | p | 6.2 | 85 | 13.6 | 22 |
| J547A | p | 22.4 | 55 | 2.5 | 22 |
| | | 140 | | | - 195 |
| J559B | p | 5.7 | 80 | 12.8 | 22 |
| | | 54 | 150 | 2.8 | - 195 |

Better contacts are needed. The present plan is to fire silver pads (to which wires may be soldered) onto the substrate before depositing the semiconductor and then connect the pad to p or n semiconductor by vacuum depositing In or Sn respectively.

The circuit elements $B_2$, D, V, and $R_6$ have been replaced by a Kiethly Model C 660 dc differential voltmeter with null adjustment and full scale readings from 0.1 mv to 500 v; $B_1$ has been replaced by an Electronic

Measurement Model C 636 constant current supply, 0.0015 ma to 220 ma. Sample geometries are shown in Figure 2.

2.3 Attempts at annealing GaAs were unsuccessful because at $700^{o}$ and $600^{o}$ the GaAs evaporated before any improvement of the film could be effected. Additional attempts will be made by covering the GaAs with a deposited film of $CaF_2$, SiO, or $SiO_2$ before annealing.

2.4 A simple ultrahigh vacuum system was built and used for deposition of germanium. (Figure 3). Five depositions were made.

In run No. 1, the system, attached to the Veeco 4 inch vacuum system, was outgassed by placing the 8" x 1" silica tube in a tube furnace and heating to about $1000^{o}$ C (temperature estimated by eye). In the tube was a fused silica supporting framework, a tungsten filament loaded with Ge, a tantalum sheet with hooks supporting a $CaF_2$ substrate, and barium getters designed for use in vacuum tube manufacture. One of two barium getters partly evaporated during the outgassing. When the pressure had dropped to $2 \times 10^{-6}$ torr, after about 14 hours of outgassing, the tube was sealed off from the Veeco system and the pressure rose temporarily to $3 \times 10^{-4}$ torr. The tube was then placed in the coil of an RF induction heater and the first getter evaporated. The pressure dropped to $1 \times 10^{-7}$. Getter No. 2 was then fired and the pressure dropped to $4 \times 10^{-8}$. The end of the tube was immersed in liquid nitrogen and

the pressure dropped to $2.4 \times 10^{-8}$. The Ge and substrate were then heated by positioning the RF coil between them, and the RF power raised gradually. The pressure rose temporarily at each increase of power until evaporation started. Temperatures were estimated by eye. The substrate heater reached $800^{\circ}$ while the Ge was at $900^{\circ}$ so the coil was moved and power increased until the Ge melted while the substrate heater was at about $600^{\circ}$. Evaporation continued for 10 minutes at a pressure of 3 to $4 \times 10^{-6}$. On cooling, the pressure was $3 \times 10^{-8}$ and rose only to $4 \times 10^{-8}$ on removing the liquid nitrogen. The deposit was polycrystalline.

In run No. 2, two grams of activated charcoal were inserted in place of the getters and a stainless steel optical baffle was placed between the Ge and the outlet to the Veeco system. The small system was outgassed at $650^{\circ}$ (thermocouple outside the silica tube) for 3 days and the attached gauge at $420^{\circ}$ for a short time until the pressure dropped to $3 \times 10^{-4}$. During seal off the pressure rose to over $10^{-3}$ but dropped, on cooling to room temperature, to $4 \times 10^{-9}$. The pressure read $6 \times 10^{-6}$ as deposition started but rose to over $1 \times 10^{-3}$ by the end of the deposition and remained above $10^{-3}$ for several hours after the power was turned off. The deposit was single crystal (Figure 4) with the familiar diffuse X.

In run No. 3, mica baffles were inserted between 1 gram of carbon, the

evaporation framework, and the attached vacuum gauge. The carbon was outgassed at $820^{o}$ while the Ge near the end of the tube furnace remained somewhat cooler. The gauge was outgassed at $450^{o}$. The pressure before seal off was $2 \times 10^{-4}$ with everything hot but, on shifting the tube in order to get at the intended seal off site, the pressure went over $10^{-3}$ so it was shifted back until the pressure dropped to $6 \times 10^{-4}$ and sealed off with the usual pressure rise to over $10^{-3}$. On cooling the pressure dropped to $4 \times 10^{-9}$. The mica had dehydrated and may have acted as a gas reservoir. The substrate heater was preheated to $900^{o}$ C and deposition carried out with the substrate heater at about $750^{o}$ and the pressure reading about $2 \times 10^{-5}$. The next morning the pressure was $1.6 \times 10^{-6}$ and the deposit was polycrystalline.

In run No. 4, Vycor was used for baffles and Ti as the getter. The tube was outgassed at $700^{o}$ - $795^{o}$ for 6 hours and at $795^{o}$ for 16 more hours, the pressure dropping to $4 \times 10^{-6}$. The system was then tipped off. On cooling the pressure dropped to $5 \times 10^{-9}$. During firing of the Ti getter, the pressure rose temporarily to $6 \times 10^{-6}$ as the silica tube near the getter got red hot. The pressure then settled at $2 \times 10^{-8}$. During evaporation the pressure could not be read because of electrical interference but was $5 \times 10^{-8}$ immediately after the RF was turned off and $6 \times 10^{-9}$ when the system had cooled down. The deposit was polycrystalline.

In run No. 5, no getter was used. The system was outgassed at 750° C for about 100 hours and tipped off. The pressure was $4 \times 10^{-9}$ but rose to $10^{-6}$ when the silica tube was flame heated to about 800° C. After cooling, the pressure settled at $2 \times 10^{-8}$. During deposition the pressure was not read but after deposition it dropped, as cooling proceeded, from $1.5 \times 10^{-7}$ to $2 \times 10^{-8}$. The deposit was partly polycrystalline but there was no detectable diffuse X.

We shall repeat the depositions in a dynamic $10^{-8}$ vacuum system which Vactite has promised to make available.

2.5 Barrier diodes and field effect transistors were constructed by Dr. Macha and Mr. Sun using $Ge/Al_2O_3$ and $Ge/CaF_2$. The work will be reported in detail in QR 3 of our contract NObsr 87634 with the Bureau of Ships and demonstrates that active devices can be made with polycrystalline films of Ge or Si.

2.6 No deposits of GaAs on BeO were made but will be when an adequate supply of BeO crystals is obtained. $CeO_2$ crystal growth will be attempted by the Société Générale Métallurgique de Hoboken, Belgium.

We are able to deposit single crystal GaAs on mica, though with some twinning and polycrystalline material, (Figure 5), but mica seems an unsatisfactory substrate because it is mechanically liable to delamination

(Figure 6) so the approach has not been pursued.

2.7 Electron microscope stereophotographs made of replicas of GaAs film surfaces demonstrated that replication with polystyrene did not show enough detail and may well have introduced artifacts due to local stretching of the polystyrene. Replications were made with evaporated carbon films on pre-shadowed surfaces. The replicas were removed by floating on $HF-HNO_3-H_2O$ which dissolves the GaAs. Shadowing has been done with Pt and with tungsten. Of four methods of evaporating platinum, soaking graphite rod in chloro-platinic acid, wrapping a graphite rod with Pt wire, evaporating Pt from between parallel, touching W wires, and evaporating Pt-C pellets between C electrodes, the last has given the thickest films and therefore best results so far, but efforts to improve the wrapped wire method will be made. Tungsten as shadowing material may be sublimed just below the melting point. A wire with a short thinned segment burned out before much W evaporated but a method using massive heat sinks consisting of pairs of half inch square copper bars two centimeters apart to clamp a 20 mil wire (see R. G. Hart, Journal of Applied Physics, Vol. 34, p. 434, 1963) worked well if the temperature of the wire was kept just below the melting point. This was done by continually adjusting the voltage to keep the space current from the hot W to the ground of the vacuum system at a few microamperes.

3.0 PLANS FOR THE SIXTH PERIOD

3.1 To deposit films in a dynamic $10^{-8}$ vacuum system.

3.2 To investigate annealing of GaAs films.

3.3 To make Hall measurements at a series of temperatures.

3.4 To investigate deposition at microns per second.

3.5 To improve the resolution in electron microscope surface studies and to investigate a setup for etching thicker films down to thicknesses suitable for electron microscopy.

3.6 To continue device studies with films of available quality.

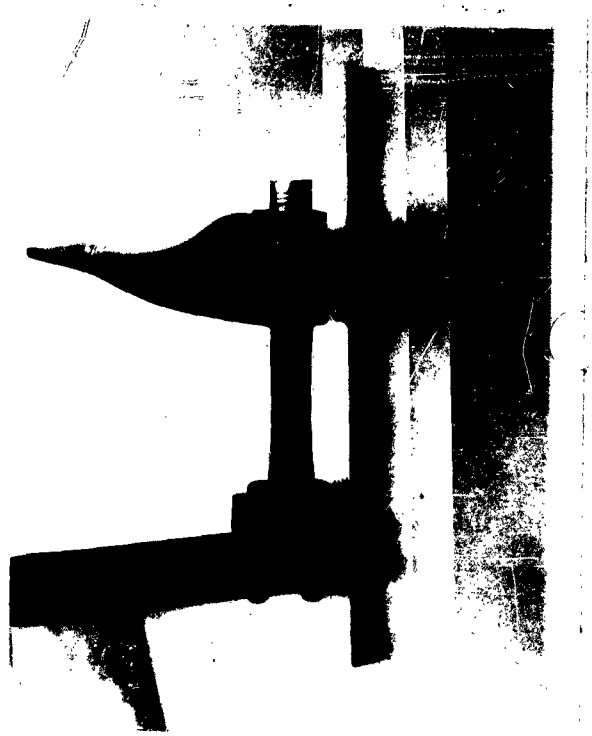

FIGURE 1

Vertical flash evaporation source

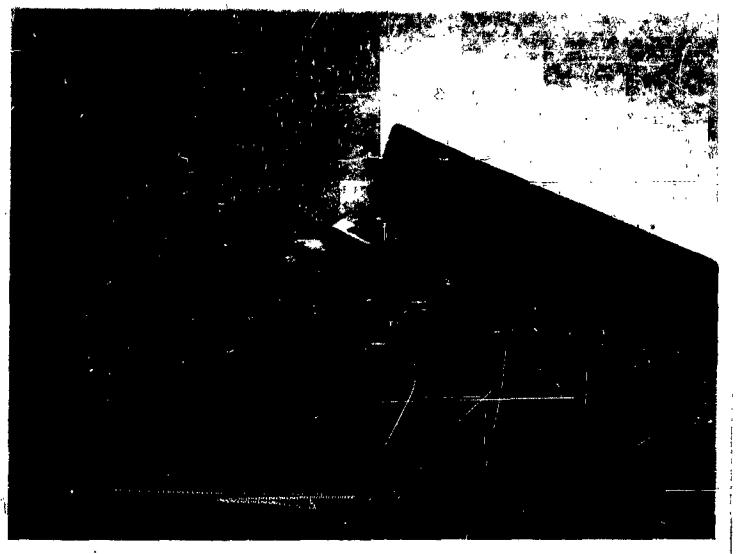

FIGURE 2

Sample structures for Hall and resistivity measurements

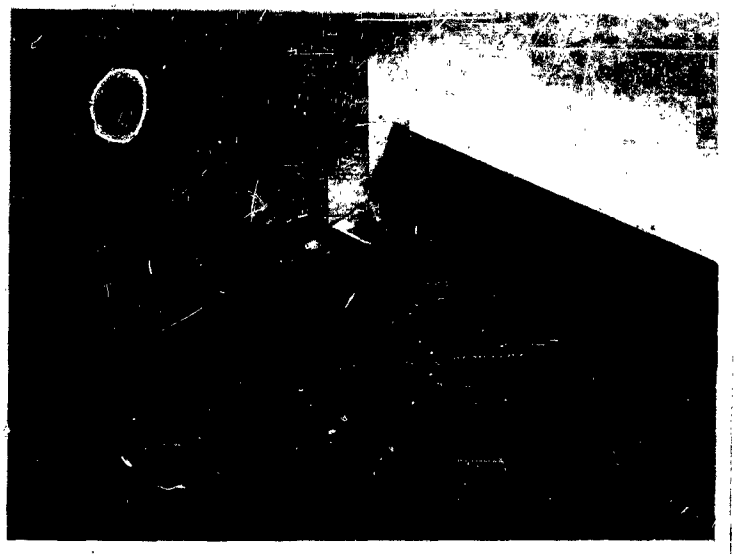

**FIGURE 2**

**Sample structures for Hall and resistivity measurements**

FIGURE 3

Glass system for deposition in higher vacuum

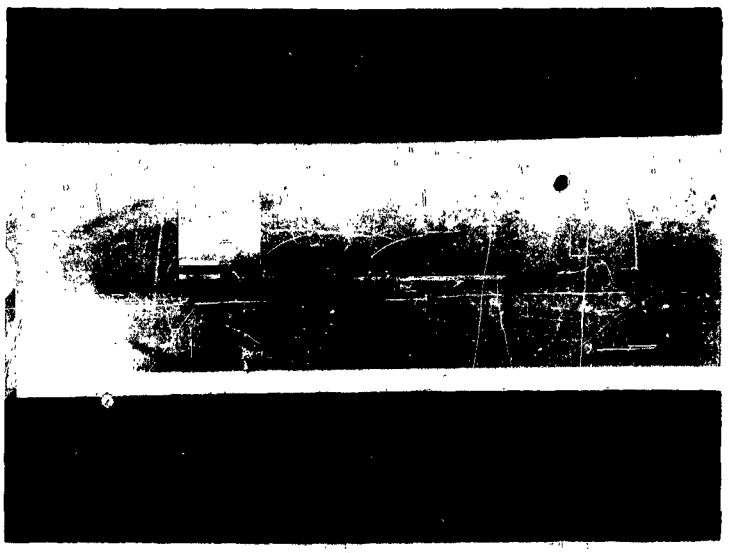

FIGURE 4

Glancing angle oscillation x-ray diffraction pattern of Ge/$CaF_2$ deposited in a system like that in Figure 3 (UHV-2, film 1710)

FIGURE 5

Glancing angle oscillation x-ray diffraction pattern of GaAs/mica (J557 A, film 1681)

FIGURE 6

Ga As/mica, J557A, 12 X